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QA/QC Review of Selected Ketone, Glycol and Diol Analysis of Groundwater from the Hooker/Ruco Superfund Site Hicksville, New York May 11, 1992

SUMMARY

Groundwater samples from six monitoring well at Hooker/Ruco Superfund Site at Hicksville, New York were analyzed for selected ketone, glycol and diol compounds. The analyses included method blanks, blank spikes, matrix spikes and matrix spike duplicates (MS/MSD). These data have been reviewed and judged to be acceptable with the following exceptions: 2-ethyl-2(hydroxymethyl-1,3-propane diol; 1,2,6-trihydroxy hexane and 2,2,4,4-tetramethyl-1,3-pentanone must be flagged estimated at concentrations below 1 mg/L.

INTRODUCTION

Groundwater samples were collected from monitoring wells P1, F1, J1, 10593, K1, and L1 on 11/06 and 11/07/92. The samples were extracted on 11/07 and 11/08/92 with methyl tert-butyl ether (MTBE) and both the MTBE extract and the remaining aqueous sample were concentrated and analyzed for the selected compounds listed in Table 1 using flame ionization gas chromatography. MTBE extract QC included two laboratory blanks, four blank spikes and one matrix spike/matrix spike duplicate. Aqueous concentrate QC included two laboratory blanks, three blank spikes, two matrix spikes and one matrix spike/matrix spike duplicate (MS/MSD).

METHODOLOGY

The analytical method for groundwater is described in Appendix 1: ***Analytical Method for the Analysis of Selected Ketone, Glycol and Diol Compounds in Groundwater, April 27, 1992.***

VALIDATION PROCEDURE

The validation procedure and results are described in Appendix 2: ***Selected Ketone, Glycol and Diol Compound Validation Study, April 27, 1992.***

SAMPLE ANALYSIS

The analysis of groundwater samples from the Hooker/Ruco Superfund Site are presented in Appendix 3: ***Analytical Results of Selected Ketone, Glycol and Diol compounds in Groundwater Samples from the Hooker/Ruco Superfund Site; Hicksville, New York, May 4, 1992.***

QA/QC REVIEW

The analytical methodology involves two fractions, the MTBE extract and the aqueous concentrate. The groundwater analysis of the MTBE extract included 2 laboratory blanks, 4 blank spikes and 1 matrix spike/matrix spike duplicate (MS/MSD). The groundwater analysis of the aqueous concentrate included 2 laboratory blanks, 3 blank spikes, 2 matrix spikes and 1 matrix spike/matrix spike duplicate (MS/MSD). Table 4 shows the laboratory blank results. Tables 5a and 5b show the blank and matrix spike results. Samples were extracted within the required holding time of 7 days.

MTBE Extract Analysis

Method Blanks

A method blank was extracted on each day that groundwater samples were extracted. Selected compounds (Table 2) were not detected in any of the method blanks. Method blanks are summarized in Table 4.

Blank Spike

Four blank spikes were prepared and analyzed at concentrations of 0.1, 0.1, 1 and 10 mg/L of each selected compound in Table 2. The percent recovery of 2,2,4,4-tetramethyl-3-pentanone at 0.1 mg/L was poor (22%). The percent recovery of dimethyl malonate ranged form 39 to 52. These recoveries, although low, are considered acceptable because of good reproducability. All other percent recoveries ranged from 42 to 107%. Acceptable recoveries for this method are 40 to 150% recovery. Results are shown in Table 5a.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD was prepared and analyzed at a concentration of 1 mg/L of each selected compound in Table 3. Sample K1 MS/MSD percent recoveries ranged from 40 to 96%. Acceptable percent recoveries for this method are 40 to 150%. Results are shown in Table 5b.

Aqueous Concentrate Analysis

Method Blanks

A method blank was extracted on each day that groundwater samples were extracted. Selected compounds (Table 3) were not detected in any of the method blanks. Method blanks are summarized in Table 4.

Blank Spike

Three blank spikes were prepared and analyzed at concentrations of 0.1, 1 and 10 mg/L of each selected compound in Table 3. Recoveries of 2-ethyl-2(hydroxymethyl)-1,3-propane diol and 1,2,6-trihydroxy hexane at 0.1 mg/L were not detected. Percent recoveries of 2-ethyl-2(hydroxymethyl)-1,3-propane diol at 1 mg/L were poor (15% and 16%). Results for 2-ethyl-2(hydroxymethyl)-1,3-propane diol have been flagged estimated (E) at concnetrations below 1 mg/L. All other percent recoveries ranged from 47 to 142%. Acceptable recoveries for this method are 40 to 150% recovery. Results are shown in Table 5b.

Matrix Spike

Two matrix spikes were prepared and analyzed at concentrations of 1 and 4 mg/L of each selected compound in Table 3. The percent recovery of 1,2,6-trihydroxy hexane at 1 mg/L for K1 Spike and 10593 Spike were 26 and 30%, respectively. These results at low concentrations should be considered suspect. Results for 1,2,6-trihydroxy hexane have been flagged estimated (E) at concentration below 1 mg/L. All other percent recoveries ranged from 57 to 139%. Results are shown in Table 5b.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD was prepared and analyzed at a concentration of 1 mg/L of each selected compound in Table 3. Sample K1 MS/MSD percent recoveries of 2-ethyl-2(hydroxymethyl-1,3-propane diol; 1,2,6-trihydroxy hexane were; and triethylene glycol were: 16 and 15%; 20 and 19%; and 36 and 35%, respectively. All other percent recoveries of sample K1 MS/MSD range from 54 to 113%. Acceptable percent

Table 1.
Selected Ketone, Glycol and Diol Compounds

2,2,4,4-tetramethyl-1,3-pentanone	1,3-butane diol
2-ethoxy ethanol	2,2-dimethyl-1,3-propane diol
2,6-dimethyl-4-heptanol	dipropylene glycol
2-ethyl-1-hexanol	1,4-butane diol
dimethyl malonate	diethylene glycol
hexanoic acid	1,6-hexane diol
2,2,4-trimethyl-1,3-pentane diol	e-caprolactam
2-ethyl hexanoic acid	triethylene glycol
heptanoic acid	cis,trans-1,4-cyclohexane dimethanol
octanoic acid	1,2,6-trihydroxyhexane
bis(2-ethylhexyl)adipate	1-methyl-2-pyrrolidinone
1,2-propane diol	ethylene glycol
2-ethyl-2(hydroxymethyl)-1,3-propane diol	2-ethoxyethyl acetate

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recoveries for this method are 40 to 150%. Results are shown in Table 5b.

Total Organic Carbon Analysis

Total organic carbon (TOC) analyses was performed on the groundwater samples on 11/11/91. The analyses included 1 blank deionized (DI) water spike, 1 matrix spike (F1) and 1 reference standard. The percent recoveries of the DI spike, matrix spike and reference standard were: 120%; 110%; and 106% respectively. The results are shown in Table 6.

CONCLUSION

The analytical data has been reviewed and the following compounds have been identified as estimated (E) at concentrations below 1 mg/L: 2-ethyl-2(hydroxymethyl-1,3-propane diol; 1,2,6-trihydroxy hexane and 2,2,4,4-tetramethyl-1,3-pentanone. These compounds were not detected in the groundwater samples at or above 0.1 mg/L, however, the values have been flagged estimated (E). These data have been judged acceptable with the additional qualifiers. See Tables 7 and 8 for updated analytical results showing qualified data.

Table 2.
MTBE Extractable Compounds

2,2,4,4-Tetramethyl-1,3-pentanone
2-Ethoxyethyl acetate
2-Ethoxy ethanol
2,6-Dimethyl-4-heptanol
2-Ethyl-1-hexanol
Dimethyl malonate
Hexanoic acid
2,2,4-Trimethyl-1,3-pentane diol
2-Ethyl hexanoic acid
Heptanoic acid
Octanoic acid
bis(2-Ethylhexyl)adipate

Table 3.
Aqueous Extract (after MTBE extraction) Compounds

1,2-propane diol
ethylene glycol
1-methyl-2-pyrrolidinone
1,3-butane diol
2,2-dimethyl-1,3-propane diol
dipropylene glycol
1,4-butane diol
diethylene glycol
1,6-hexane diol
e-caprolactam
triethylene glycol
cis,trans-1,4-cyclohexane dimethanol
2-ethyl-2(hydroxymethyl)-1,3-propane diol
1,2,6-trihydroxyhexane

Table 4.
Method Blank Results

Parameter	Date	Laboratory Blank ug/mL 11/07/91	Laboratory Blank ug/mL 11/08/91
MTBE Extract			
2,2,4,4-tetramethyl-3-pentanone		ND 0.1	ND 0.1
2,6-dimethyl-4-heptanol		ND 0.1	ND 0.1
2-ethyl-1-hexanol		ND 0.1	ND 0.1
dimethyl malonate		ND 0.1	ND 0.1
hexanoic acid		ND 0.1	ND 0.1
2,2,4-trimethyl-1,3-pentane diol		ND 0.1	ND 0.1
2-ethyl-hexanoic acid		ND 0.1	ND 0.1
heptanoic acid		ND 0.1	ND 0.1
octanoic acid		ND 0.1	ND 0.1
bis(2-ethylhexyl) adipate		ND 0.1	ND 0.1
Aqueous Concentrate			
2,2-dimethyl-1,3-propane diol		ND 0.1	ND 0.1
1,2-propane diol		ND 0.1	ND 0.1
ethylene glygol		ND 0.1	ND 0.1
1-methyl-2-pyrrolidinone		ND 0.1	ND 0.1
1,3-butane diol		ND 0.1	ND 0.1
2,2-dimethyl-1,3-propane diol		ND 0.1	ND 0.1
dipropylene glycol		ND 0.1	ND 0.1
1,4-butane diol		ND 0.1	ND 0.1
diethylene glycol		ND 0.1	ND 0.1
1,6-hexane diol		ND 0.1	ND 0.1
e-caprolactam		ND 0.1	ND 0.1
triethylene glycol		ND 0.1	ND 0.1
cis/trans-1,4-cyclohexane dimethanol		ND 0.1	ND 0.1
cis/trans-1,4-cyclohexane dimethanol		ND 0.1	ND 0.1
2-ethyl-2(hydroxymethyl)-1,3-propane diol		ND 0.1	ND 0.1
1,2,6-trihydroxy hexane		ND 0.1	ND 0.1

ND x is defined as not detected at or above x.

Table 5a.
MTBE Extract
Spike Recovery Data
Percent Recovery (%)

Parameter	Blank Spike 0.1 ppm	Blank Spike 10 ppm	K1 Spike 1 ppm	K1 Spike 1 ppm	Blank Spike 0.1 ppm	Blank Spike 1 ppm
2,2,4,4-tetramethyl-3-pentanone	42	76	49	46	22	66
2,6-dimethyl-4-heptanol	73	92	75	73	60	79
2-ethyl-1-hexanol	82	92	79	77	69	81
dimethyl malonate	48	52	41	40	39	46
hexanoic acid	107	91	81	82	100	91
2,2,4-trimethyl-1,3-pentane diol	59	87	57	61	56	72
2-ethyl-hexanoic acid	60	91	82	86	57	76
heptanoic acid	66	88	77	81	60	74
octanoic acid	69	88	81	92	63	75
bis(2-ethylhexyl) adipate	89	100	85	96	88	92

Table 5b.
Aqueous Concentrate
Spike Recovery Data
Percent Recovery (%)

Parameter	Blank Spike 0.1 ppm	Blank Spike 10 ppm	K1 Spike 1 ppm	K1 Spike 1 ppm	Blank Spike 1 ppm	K1 Spike 4 ppm	K1 Spike 1 ppm	10593 Spike 1 ppm
1,2-propane diol	89	101	80	82	110	116	133	117
ethylene glygol	97	104	67	71	123	139	151	152
1-methyl-2-pyrrolidinone	84	96	80	81	91	94	96	80
1,3-butane diol	83	95	74	81	100	104	110	94
2,2-dimethyl-1,3-propane diol	93	106	81	89	90	105	138	117
dipropylene glycol	86	102	73	78	142	124	116	112
1,4-butane diol	88	95	103	113	101	106	100	96
diethylene glycol	95	90	98	97	74	82	93	119
1,6-hexane diol	80	92	72	70	90	99	85	80
e-caprolactam	82	94	66	62	95	96	77	78
triethylene glycol	106	91	36	35	106	152	88	80
cis/trans-1,4-cyclohexane dimethanol	70	76	55	55	107	105	88	99
cis/trans-1,4-cyclohexane dimethanol	76	79	54	54	110	109	72	67
2-ethyl-2(hydroxymethyl)-1,3-propane diol	0	50	16	15	92	84	57	57
1,2,6-trihydroxy hexane	0	47	20	19	121	114	26	30

Table 6.
Total Organic Carbon
Spike Recovery Data

Identification	Amount Added mg/L	Amount Found mg/L	Spike Recovery %
Blank Spike	1000	1200	120%
Sample F1 Spike	1	1.1	110%
Reference Standard	400	420	106%

Table 7.
MTBE Extract
Analytical Results
mg/L
Qualified Data

Parameter	P1	F1	J1	10593	K1	L1
Date	11/06/91	11/06/91	11/06/91	11/07/91	11/07/91	11/07/91
2,2,4,4-tetramethyl-3-pentanone	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)
2,6-dimethyl-4-heptanol	ND 0.1	1.3	0.1	ND 0.1	ND 0.1	ND 0.1
2-ethyl-1-hexanol	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
dimethyl malonate	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
hexanoic acid	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
2,2,4-trimethyl-1,3-pentane diol	0.2	1.1	0.3	ND 0.1	ND 0.1	ND 0.1
2-ethyl-hexanoic acid	0.4	4.0	0.1	ND 0.1	ND 0.1	ND 0.1
heptanoic acid	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
octanoic acid	ND 0.1	ND 0.1	ND 0.1	ND 0.1	0.1	ND 0.1
bis(2-ethylhexyl) adipate	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
2,2-dimethyl-1,3-propane diol	0.5	31	1.5	0.3	0.1	ND 0.1

ND x is defined as not detected at or above x.

Table 8.
Aqueous Concentrate
Analytical Results
mg\L
Qualified Data

Parameter	P1	F1	J1	10593	K1	L1
Date	11/06/91	11/06/91	11/06/91	11/07/91	11/07/91	11/07/91
1,2-propane diol	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
ethylene glygol	ND 0.1	ND 0.1	0.1	0.1	0.1	ND 0.1
1-methyl-2-pyrrolidinone	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
1,3-butane diol	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
2,2-dimethyl-1,3-propane diol	5.3	190	4.9	4.3	2.1	0.1
dipropylene glycol	ND 0.1	ND 0.1	ND 0.1	0.1	ND 0.1	ND 0.1
1,4-butane diol	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
diethylene glycol	ND 0.1	ND 0.1	ND 0.1	0.3	0.2	0.1
1,6-hexane diol	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
e-caprolactam	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
triethylene glycol	ND 0.1	ND 0.1	ND 0.1	0.2	0.1	0.1
cis/trans-1,4-cyclohexane dimethanol	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
cis/trans-1,4-cyclohexane dimethanol	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
2-ethyl-2(hydroxymethyl)-1,3-propane diol	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)
1,2,6-trihydroxy hexane	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)	ND 0.1 (E)

ND x is defined as not detected at or above x.

QA/QC Review
of Method Validation for
the Analysis of Selected
Ketone, Glycol and Diol Compounds
May 4, 1992

INTRODUCTION

A method validation for determining selected ketone, glycol and diol compounds in groundwater at a method detection limit of 100 ug/L has been reviewed. The validation consisted of three matrix spike levels extracted and analyzed in triplicate. The extracts were analyzed for selected ketone, glycol, and diol compounds (Tables 1 and 2) using GC/FID. Table 1 shows the compounds extracted by methyl tert-butyl ether (MTBE). Table 2 shows compounds that are concentrated in the aqueous phase, after MTBE extraction. These data have been reviewed and judged acceptable with the following exceptions: 2,2,4,4-tetramethyl-1,3-pentanone and 2,2,4-trimethyl-1,3-pentane diol must be flagged estimated at concentrations below 1 mg/L and 2-ethoxyethyl acetate has been judged unacceptable.

METHODOLOGY

The analytical method for ground water is described in Appendix 1: ***Analytical Method for the Analysis of Selected Ketone, Glycol and Diol Compounds in Ground Water, April 27, 1992.***

VALIDATION PROCEDURE

The validation procedure and results are described in Appendix 2: ***Selected Ketone, Glycol and Diol Compound Validation Study, April 27, 1992.***

QA/QC REVIEW

The validation procedure included 5 method blanks and 15 matrix spike analyses results. Tables 3a and 3b show the results of the matrix spike samples.

Matrix Spikes

Samples were spiked at 0.1, 1, and 10 mg/L of each of the selected ketone, glycol and diol compounds listed in Tables 1 and 2. MTBE extract percent recoveries, mean percent recoveries, and percent relative standard deviations are shown in Table 3a. Aqueous concentrate percent recoveries, mean percent recoveries, and percent relative standard deviations are shown in Table 3b.

MTBE Extract: Method accuracy is measured by percent recovery. The mean percent recoveries ranged from 4-126%. Acceptable percent recovery limits for this method are 60-130%. Validation recoveries for 2-ethoxyethyl acetate (4%) and 2,2,4-trimethyl-1,3-pentane diol (34%) were below the acceptable limits. This method is not suitable for 2-ethoxyethyl acetate based on recovery performance. The method performance for 2,2,4-trimethyl-1,3-pentane diol is variable, based on recovery fluctuations. This method is capable of extracting and analyzing for 2,2,4-trimethyl-1,3-pentane diol in ground water, however, matrix spike recoveries must be reviewed to verify extraction and analysis efficiency. Acceptable percent recoveries for all other compounds listed in Table 1 are within acceptable limits.

Method precision is measured by relative percent deviation (%RSD). The %RSD ranged from 9.6-29%. Acceptable %RSD values are below 20%. Two compounds, 2,2,4,4-tetramethyl-1,3-pentanone (29%) and hexanoic acid (21%) were reported with %RSD values greater than 20%. The percent recoveries for 2,2,4-trimethyl-1,3-pentane diol were variable and contained 2 values below the detection limit, therefore, no %RSD was calculated. These results indicate that spike recoveries for these compounds are variable. Percent recoveries of these compounds should be closely monitored. Validation %RSD values for all other

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compounds listed in Table 1 are within acceptable limits.

Aqueous Concentrate: Method accuracy is measured by percent recovery. The mean percent recoveries ranged from 85-120%. Acceptable percent recovery limits for this method are 60-130%. Validation percent recoveries for all compounds shown in Table 2 are within acceptable limits.

Method precision is measured by relative percent deviation (%RSD). The %RSD ranged from 3.7-12%. Acceptable %RSD values are below 20%. Validation %RSD values for all compounds shown in Table 2 are with acceptable limits.

CONCLUSION

The method validation data has been reviewed. The method performance for all compounds, other than 2-ethoxyethyl acetate; 2,2,4,4-tetramethyl-1,3-pentanone; and 2,2,4-trimethyl-1,3-pentane diol listed in Tables 1 and 2 are judged acceptable. The method performance for 2-ethoxyethyl acetate has been judged unacceptable. The method performance for 2,2,4,4-tetramethyl-1,3-pentanone and 2,2,4-trimethyl-1,3-pentane diol are poor at low concentrations and should be flagged estimated at concentrations below 1 mg/L.

Table 1.
MTBE Extractable Compounds

2,2,4,4-Tetramethyl-1,3-pentanone
2-Ethoxyethyl acetate
2-Ethoxy ethanol
2,6-Dimethyl-4-heptanol
2-Ethyl-1-hexanol
Dimethyl malonate
Hexanoic acid
2,2,4-Trimethyl-1,3-pentane diol
2-Ethyl hexanoic acid
Heptanoic acid
Octanoic acid
bis(2-Ethylhexyl)adipate

Table 2.
Aqueous Concentrate Compounds

1,2-propane diol
ethylene glycol
1-methyl-2-pyrrolidinone
1,3-butane diol
2,2-dimethyl-1,3-propane diol
dipropylene glycol
1,4-butane diol
diethylene glycol
1,6-hexane diol
e-caprolactam
triethylene glycol
cis,trans-1,4-cyclohexane dimethanol
2-ethyl-2(hydroxymethyl)-1,3-propane diol
1,2,6-trihydroxyhexane

Table 3a.
Matrix Spike Percent Recovery
and Percent Relative Deviations
for MTBE Extractable Compounds

Parameter	Percent Recovery									Mean	%
Spike	0.1 ppm			1 ppm			10 ppm				
Run	1	2	3	1	2	3	1	2	3	% Rec	RSD
2,2,4,4-tetramethyl-3-pentanone	36	42	35	63	53	59	74	81	81	58	29
2-ethoxethyl acetate	0	0	0	6	6	6	6	7	6	4	NC
2-ethoxy ethanol	65	81	88	74	75	74	71	76	75	76	8.0
2,6-dimethyl-4-heptanol	79	98	97	94	100	97	110	114	110	100	10
2-ethyl-1-hexanol	94	114	122	103	110	108	116	121	119	112	8.5
dimethyl malonate	56	69	76	63	63	62	61	66	65	65	8.1
hexanoic acid	76	54	59	82	97	97	100	120	106	100	21
2,2,4-trimethyl-1,3-pentane diol	58	0	0	70	62	51	23	24	21	34	NC
2-ethyl hexanoic acid	100	94	107	89	107	105	107	124	112	107	9.6
heptanoic acid	97	85	97	88	102	101	104	123	109	105	11
octanoic acid	113	104	123	88	107	105	107	130	113	109	11
bis-(2-ethylhexyl) adipate	109	115	115	123	137	135	128	139	136	126	11

NC is defined as not calculated.

Table 3b.
Matrix Spike Percent Recovery
and Percent Relative Deviations
for Aqueous Concentrate Compounds

Parameter	Spike	Percent Recovery									Mean	%
		0.1 ppm			1 ppm			10 ppm				
	Run	1	2	3	1	2	3	1	2	3	% Rec	RSD
1,2-propane diol		86	83	96	110	105	115	99	98	100	99	9.6
ethylene glygol		80	87	79	110	100	112	96	95	97	95	12
1-methyl-2-pyrrolidinone		105	98	109	99	103	107	96	94	97	101	5.0
1,3-butane diol		97	91	101	112	112	116	99	97	101	103	7.8
2,2-dimethyl-1,3-propane diol		96	101	102	93	92	97	87	85	90	94	5.8
dipropylene glycol		87	95	97	116	113	117	115	103	103	105	11
1,4-butane diol		91	99	97	120	114	119	111	107	111	108	9.5
diethylene glycol		82	88	84	112	106	115	104	102	104	100	12
1,6-hexane diol		87	93	93	109	104	110	101	97	102	100	7.2
e-caprolactam		102	113	107	107	101	107	103	101	103	105	3.7
triethylene glycol		94	98	88	111	103	113	109	104	109	103	8.0
cis/trans-1,4-cyclohexane dimethanol		91	94	96	83	80	83	82	79	81	85	7.1
cis/trans-1,4-cyclohexane dimethanol		102	105	104	87	84	87	84	82	85	91	10
2-ethyl-2(hydroxymethyl)-1,3-propane diol		0	0	0	121	117	129	115	115	120	120	4.9
1,2,6-trihydroxy hexane		0	0	0	91	89	101	102	104	108	100	7.4

NC is defined as not calculated.

Appendix 1

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Analytical Method for Analysis of Selected Ketone, Glycol, and Diol Compounds in Groundwater
April 27, 1992

SCOPE AND APPLICATION

This method is used to determine the concentration of selected ketone, glycol, and diol compounds in groundwater (Table 1). The method detection limit determined for all the compounds shown in Table 1 from reagent water is 100 ug/L.

SUMMARY OF METHOD

This method provides liquid/liquid extraction procedures and gas chromatographic conditions for the detection of selected ketone, glycol and diol compounds (Table 1). SW-846 3rd Edition Method 3510 (Separatory Funnel Liquid-Liquid Extraction) is used to extract compounds listed in Table 2 using methyl tert-butyl ether (MTBE) from ground water samples. The MTBE extract is concentrated using a rotory evaporator to less than 10 mL and the final volume is adjusted to 10 mL with MTBE. A 3 uL aliquot is injected onto a gas chromatograph equipped with a acid modified polyethylene glycol (DB-FFAP) mega-bore capillary column and a flame ionization detector (GC/FID). After MTBE solvent extraction, the remaining aqueous sample is concentrated to 10 mL using a rotary evaporator under vacuum. A 3 uL aliquot of the concentrated aqueous sample is injected into a gas chromatograph equipped with a polyethylene glycol (DB-

WAX) mega-bore capillary column and a flame ionization detector (GC/FID).

APPARATUS AND MATERIALS

Gas Chromatograph:

Gas Chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases and syringes. A data system for measuring peaks heights and/or areas is also used.

Column: For analyses of MTBE extract, a DB-FFAP (acid modified polyethylene glycol) fused silica mega-bore capillary column, 30 m x 0.53 mm i.d., 1.0 um film thickness (J&W Scientific, or equivalent). For analyses of aqueous concentrate, a DB-WAX (polyethylene glycol) fused silica mega-bore capillary column, 30 m x 0.53 mm i.d., 1.0 um film thickness (J&W Scientific, or equivalent).

Detector: Flame ionization detector (FID).

Rotary-Evaporator:

A rotary-evaporator capable of maintaining a water bath temperature of 55°C and a vacuum efficiency to concentrate aqueous sample.

Laboratory Glassware:

Volumetric Flasks: 10, 50, and 100 mL ground-glass stopper.

Separatory Funnel: 2-Liter with teflon stopper

Round Bottom Flask: 1-Liter

Microsyringe:

10 uL.

Solvents:

Methyl tert-butyl ether (MTBE) and methanol pesticide grade (or equivalent).

Calibration Standards:

Calibration standards at a minimum of five concentration levels of compounds listed in Table 1 are prepared through dilution of the stock standards with methanol. Initially, a five point calibration will be analyzed to verify linearity. After linearity has been demonstrated, a three point calibration will be performed prior to analysis. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

a 20-50% methanol:water solution. The methanol/water extract is stored at 4°C prior to analysis. Figure 1 shows a flow diagram of the analytical method.

Gas Chromatography Conditions for MTBE Extract:

Column: Set the carrier gas (helium) linear velocity at 20 cm/sec. Column temperature is set to 60°C initially. The initial temperature should be maintained for 1 minute and then ramped at a rate of 4°C/minute to a final temperature of 230°C. The final temperature should be held for at least 15 minutes. The complete oven profile is given in Table 6.

Calibration for MTBE Extract:

Five concentration levels of semi-volatile compounds (Table 2) must be used for initial calibration of the GC system. The initial calibration curve is comprised of 2.5, 5, 10, 50, and 100 ug/mL concentrations. Once linearity is established, a three point calibration consisting of a 2.5, 10, 100 ug/mL standards will be used.

Gas Chromatographic Analysis of MTBE Extracts:

The elution times of the semi-volatile compounds are shown in Table 4. Figure 2 shows a sample of a GC chromatogram. The sample peak must be within +/- 0.05 minutes to be considered as positive identification. The sample area response of analyte must be within the calibration range. If the area is above the calibration range, a dilution must be performed and re-analyzed.

Calculations are performed using linear regression analysis. The area response is

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plotted versus analyte concentration using first order regression. The expression $y=mx+b$ is used to calculate sample concentration by:

$$x = (y-b)/m$$

where x is defined as the concentration in ug/L
y is defined as the area response
b is defined as the y-intercept of the calibration curve
m is defined as the slope of the calibration curve

Gas Chromatography Conditions for Aqueous Extract:

Column: Set the carrier gas (helium) linear velocity at 20 cm/sec. Column temperature is set to 60°C initially. The initial temperature should be maintained for 1 minute and then ramped at a rate of 4°C/minute to a final temperature of 220°C. The final temperature should be held for at least 15 minutes. The complete oven profile is given in Table 7.

Calibration for Aqueous Extract:

Five concentration levels of semi-volatile compounds (Table 3) must be used for initial calibration of the GC system. The initial calibration curve is comprised of 2.5, 5, 10, 50, and 100 ug/mL concentrations. Once linearity is established, a three point calibration consisting of a 2.5, 10, 100 ug/mL will be used.

Gas Chromatographic Analysis of Aqueous Extracts:

The elution times of the semi-volatile compounds are shown in Table 5. Figure 3

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shows a sample of a GC chromatogram. The sample peak must be within +/- 0.05 minutes to be considered as a positive identification. The sample area response of analyte must be within the calibration range. If the area is above the calibration range, a dilution must be performed and re-analyzed.

Calculations are performed using linear regression analysis. The area response is plotted versus analyte concentration using first order regression. The expression $y=mx+b$ is used to calculate sample concentration by:

$$x = (y-b)/m$$

where x is defined as the concentration in ug/L
y is defined as the area response
b is defined as the y-intercept of the calibration curve
m is defined as the slope of the calibration curve

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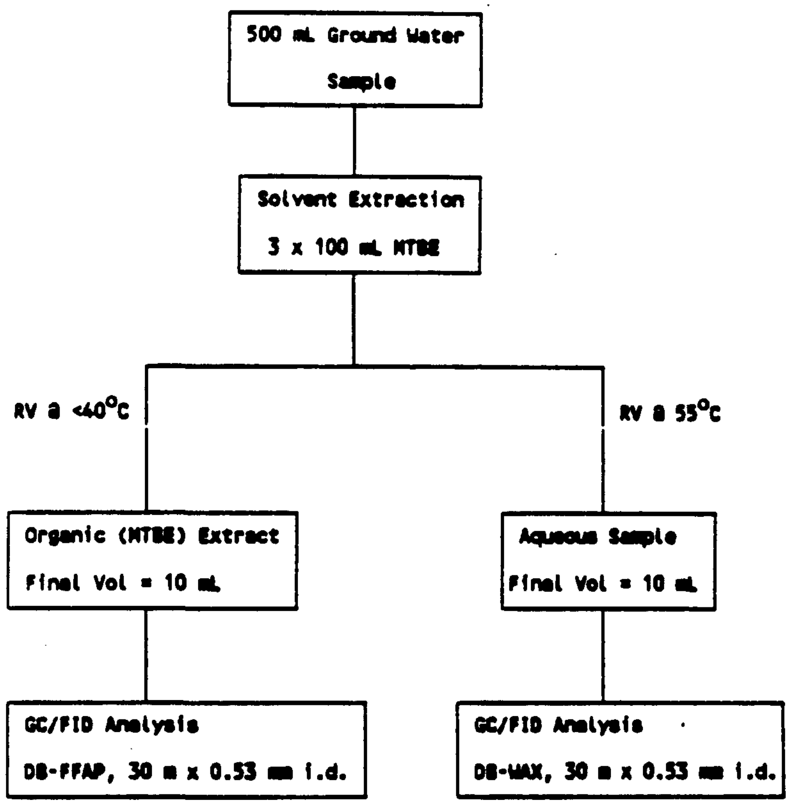
Figure 1.

FLOW DIAGRAM OF ANALYTICAL METHOD



2,2,4,4-tetramethyl-3-pentanone
2-ethoxy ethanol
2,6-dimethyl-4-heptanol
2-ethyl-1-hexanol
dimethyl malonate
hexanoic acid
2,2,4-trimethyl-1,3-pentane diol
2-ethyl hexanoic acid
heptanoic acid
octanoic acid
bis(2-ethylhexyl)adipate

1,2-propane diol
ethylene glycol
1-methyl-2-pyrrolidinone
1,3-butane diol
2,2-dimethyl-1,3-propanediol
dipropylene gylcol
1,4-butane diol
diethylene glycol
1,6-hexane diol
e-caprolactam
triethylene glycol
1,2,6-trihydroxy hexane
2-ethyl-2(hydroxymethyl)-1,3-propane diol
cis,trans-cyclohexane-
dimethanol

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Figure 2.
Gas Chromatogram of
MTBE Extract

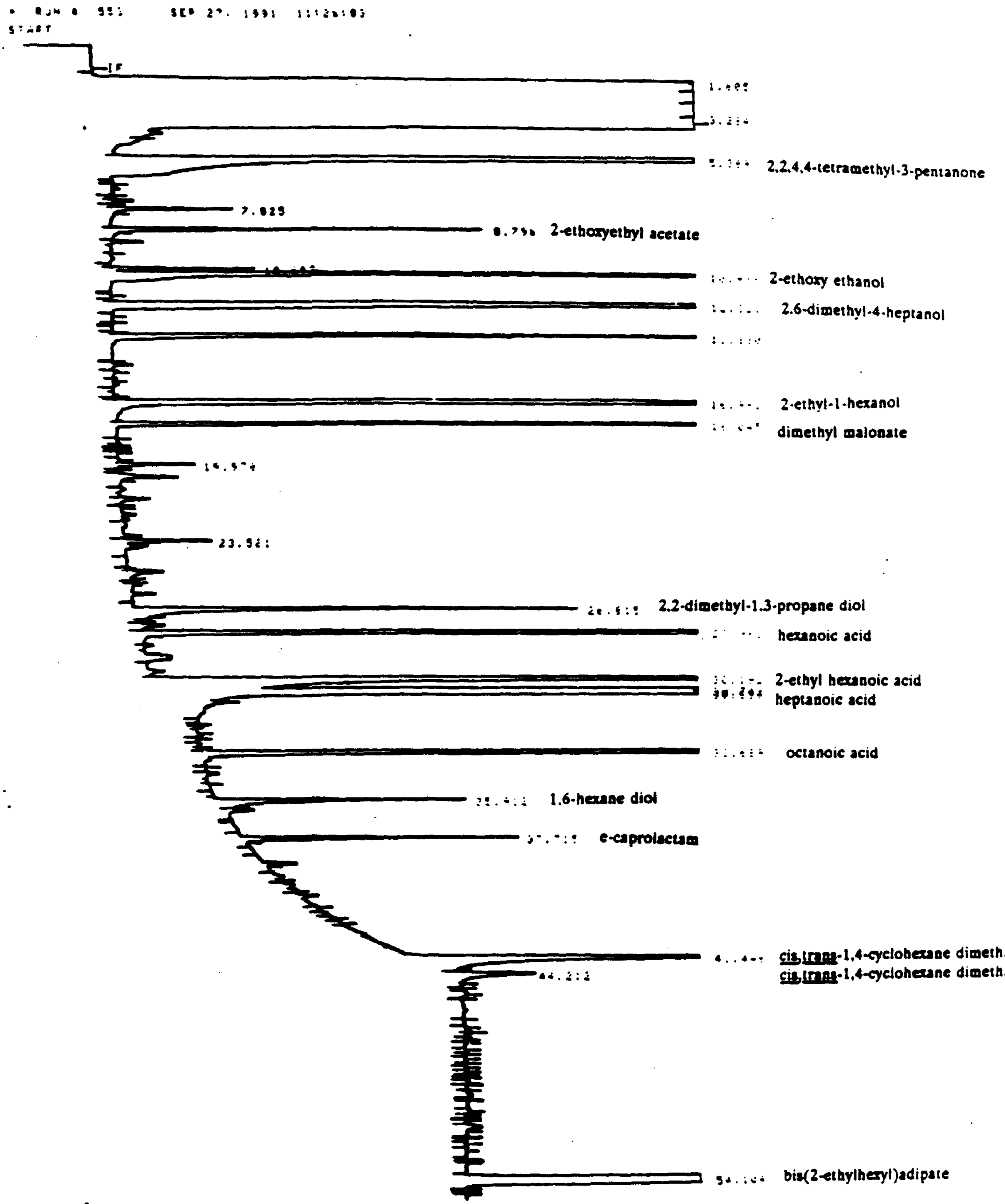

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Figure 3.
Gas Chromatogram of
Aqueous (after MTBE Extraction) Extract

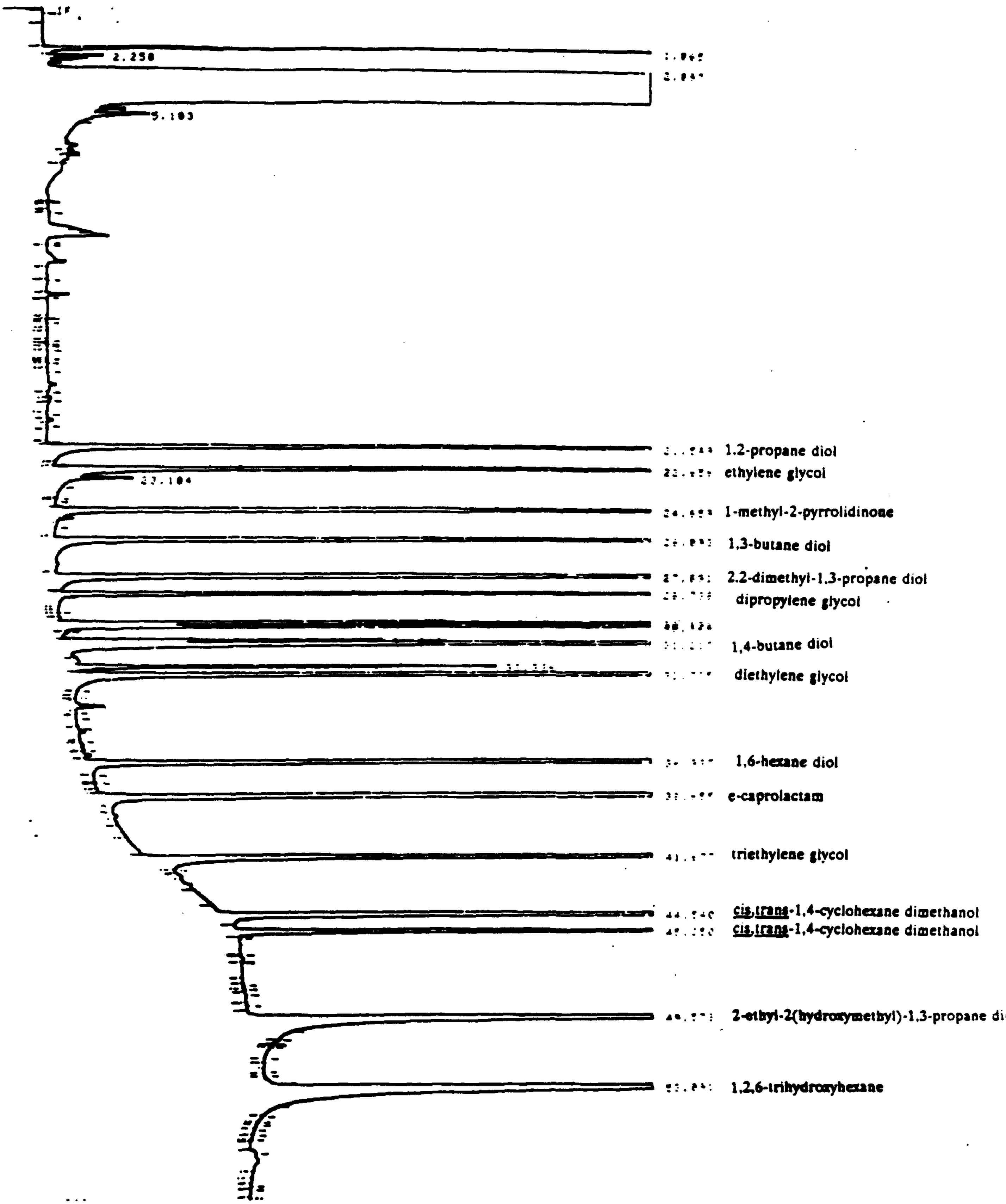

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Table 1.
Selected Ketone, Glycol and Diol Compounds

2,2,4,4-tetramethyl-1,3-pentanone	1,3-butane diol
2-ethoxy ethanol	2,2-dimethyl-1,3-propane diol
2,6-dimethyl-4-heptanol	dipropylene glycol
2-ethyl-1-hexanol	1,4-butane diol
dimethyl malonate	diethylene glycol
hexanoic acid	1,6-hexane diol
2,2,4-trimethyl-1,3-pentane diol	e-caprolactam
2-ethyl hexanoic acid	triethylene glycol
heptanoic acid	cis,trans-1,4-cyclohexane dimethanol
octanoic acid	1,2,6-trihydroxyhexane
bis(2-ethylhexyl)adipate	1-methyl-2-pyrrolidinone
1,2-propane diol	ethylene glycol
2-ethyl-2(hydroxymethyl)-1,3-propane diol	2-ethoxyethyl acetate

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Table 2.
MTBE Extractable Compounds

2,2,4,4-Tetramethyl-1,3-pentanone
2-Ethoxyethyl acetate
2-Ethoxy ethanol
2,6-Dimethyl-4-heptanol
2-Ethyl-1-hexanol
Dimethyl malonate
Hexanoic acid
2,2,4-Trimethyl-1,3-pentane diol
2-Ethyl hexanoic acid
Heptanoic acid
Octanoic acid
bis(2-Ethylhexyl)adipate

Table 3.
Aqueous Extract (after MTBE extraction) Compounds

1,2-propane diol
ethylene glycol
1-methyl-2-pyrrolidinone
1,3-butane diol
2,2-dimethyl-1,3-propane diol
dipropylene glycol
1,4-butane diol
diethylene glycol
1,6-hexane diol
e-caprolactam
triethylene glycol
cis,trans-1,4-cyclohexane dimethanol
2-ethyl-2(hydroxymethyl)-1,3-propane diol
1,2,6-trihydroxyhexane

Table 4.
MTBE Extractable Compounds
GC Retention Times

Analyte	Retention Time (min)
2,2,4,4-Tetramethyl-1,3-pentanone	5.4
2-Ethoxyethyl acetate	8.8
2-Ethoxy ethanol	10.9
2,6-Dimethyl-4-heptanol	12.3
2-Ethyl-1-hexanol	17.0
Dimethyl malonate	18.0
1-Methyl-2-pyrrolidinone	23.7
2,2-Dimethyl-1,3-propane diol	26.8
Hexanoic acid	28.0
2,2,4-Trimethyl-1,3-pentane diol	30.2
2-Ethyl hexanoic acid	30.7
Heptanoic acid	30.9
Octanoic acid	33.7
e-Caprolactam	37.7
cis,trans-1,4-cyclohexane dimethanol	43.3
bis(2-Ethylhexyl)adipate	54.1

Table 5.
Aqueous Extract (after MTBE extraction) Compounds

Analyte	Retention Time (min)
1,2-propane diol	21.5
ethylene glycol	22.6
1-methyl-2-pyrrolidinone	24.6
1,3-butane diol	26.0
2,2-dimethyl-1,3-propane diol	27.8
dipropylene glycol	28.7, 30.1, 30.3
1,4-butane diol	31.1
diethylene glycol	32.7
1,6-hexane diol	36.9
e-caprolactam	38.6
triethylene glycol	41.6
cis,trans-1,4-cyclohexane dimethanol	44.4, 45.3
2-ethyl-2(hydroxymethyl)-1,3-propane diol	49.4
1,2,6-trihydroxyhexane	53.0

Table 6.
Gas Chromatograph Conditions
for MTBE Extract Analysis

Gas Chromatograph	Hewlett-Packard Model 5880 with Model 3396 Integrator
Detector	Flame Ionization Detector (FID)
Column	DB-FFAP (polyethylene glycol - acid modified) fused silica capillary, 30 m x 0.53 i.d., 1.0 um film thickness
Injection Volume	3 uL (splitless)
Injection Port Liner	Uniliner (Restek)
Carrier Gas	Helium
Linear Velocity	18 cm/sec (head pressure 4 psi)
Initial Temperature	60°C
Initial Time	1 minute
Oven Temperature Rate	4°C/minute
Final Temperature	230°C
Final Temperature Hold	15 minutes
Detector Temperature	250°C
Detector Makeup Gas	Nitrogen
Makeup Gas Flow Rate	30 ml/minute
Detector Attenuation	Attn 2 ↑ 0

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Table 7.
Gas Chromatograph Conditions
for Aqueous (after MTBE Extraction) Analysis

Gas Chromatograph	Hewlett-Packard Model 5880 with Model 3396 Integrator
Detector	Flame Ionization Detector (FID)
Column	DB-WAX (polyethylene glycol) fused silica capillary, 30 m x 0.53 i.d., 1.0 um film thickness
Injection Volume	3 uL (splitless)
Injection Port Liner	Uniliner (Restek)
Carrier Gas	Helium
Linear Velocity	18 cm/sec (head pressure 4 psi)
Initial Temperature	60°C
Initial Time	1 minute
Oven Temperature Rate	4°C/minute
Final Temperature	220°C
Final Temperature Hold	15 minutes
Detector Temperature	250°C
Detector Makeup Gas	Nitrogen
Makeup Gas Flow Rate	30 ml/minute
Detector Attenuation	Attn 2 ↑ 0

Appendix 2

HKR 001 1787

Selected Ketone, Glycol, and Diol Compound Validation Study
April 27, 1992

SUMMARY

A gas chromatography method has been developed to analyze selected ketone, glycol, and diol compounds (Table 1) in groundwater at a method detection limit of 100 ug/L. To demonstrate the efficiency of this method, a validation has been performed. Three deionized water spikes at concentrations of 0.1, 1, and 10 mg/L of each compound listed in Table 1 were prepared and analyzed in triplicate. Twelve of the selected ketone, glycol, and diol compounds (Table 2) were extracted with methyl tert-butyl ether (MTBE). The remaining compounds (Table 3) were analyzed directly from the aqueous concentrate.

Selected ketone, glycol, and diol were extracted first from the deionized water by liquid/liquid extraction with MTBE using a separatory funnel. The MTBE extract was analyzed with a gas chromatograph (GC) equipped with a acid modified polyethylene glycol (DB-FFAP) mega-bore capillary column and a flame ionization detector (FID).

The remaining MTBE extracted aqueous sample was concentrated and analyzed using a gas chromatograph equipped with a polyethylene glycol (DB-WAX) mega-bore capillary column and a flame ionization detector (FID).

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INTRODUCTION

A validation study for selected ketone, glycol, and diol in groundwater at 100, 1000, and 10000 ug/L has been performed using gas chromatography.

METHOD

The three spike levels chosen were: 100, 1000, and 10000 ug/L of selected ketone, glycol, and diol compounds (Table 1) in water. Spikes were prepared in triplicate by adding the spike compounds to 500 mL of deionized water in a separatory funnel. Extraction was performed by SW-846, 3rd Edition Method 3510 (separatory funnel liquid-liquid extraction) with 3 x 100 mL methyl tert-butyl ether (MTBE).

The MTBE was rotary evaporated to less than 10 mL. The extract was transferred to a 10 mL volumetric flask and brought to volume with MTBE. A 3 uL aliquot of the MTBE extract was injected onto a gas chromatograph (GC) equipped with a acid modified polyethylene glycol (DB-FFAP) mega-bore capillary column and a flame ionization detector (FID). GC conditions are shown in Table 4.

The MTBE extracted aqueous sample was rotary evaporated to less that 10 mL. The aqueous extract was transferred to another 10 mL volumetric flask and brought to volume with methanol. A 3 uL aliquot of the aqueous/methanol extract was injected onto a gas chromatograph equipped with a polyethylene glycol (DB-WAX) mega-bore capillary column and a flame ionization detector (FID). GC conditions are shown in Table 5.

RESULTS

The Method Detection Limit (MDL) of 100 ug/L was determined for selected ketone, glycol and diol compounds based on chromatographic performance and signal to noise ratios. The analytical results are shown in Tables 6 and 7. The spike recoveries of 2-ethoxyethyl acetate in the MTBE extract were poor (0-7%). The spike recoveries for 2,2,4,4-tetramethyl-1,3-pentanone and 2,2,4-trimethyl-1,3pentane diol were inconsistent, possibly due to degradation within the sample or gas chromatograph. Mean spike recoveries for the remaining MTBE extractable compounds ranged from 65-126% with %RSD ranging from 8 to 11. Mean spike recoveries for the MTBE extracted aqueous concentrates ranged from 85-120% with %RSD ranging from 3.7 to 12.

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Table 1.
Selected Ketone, Glycol and Diol Compounds

2,2,4,4-tetramethyl-1,3-pentanone	1,3-butane diol
2-ethoxy ethanol	2,2-dimethyl-1,3-propane diol
2,6-dimethyl-4-heptanol	dipropylene glycol
2-ethyl-1-hexanol	1,4-butane diol
dimethyl malonate	diethylene glycol
hexanoic acid	1,6-hexane diol
2,2,4-trimethyl-1,3-pentane diol	e-caprolactam
2-ethyl hexanoic acid	triethylene glycol
heptanoic acid	cis,trans-1,4-cyclohexane dimethanol
octanoic acid	1,2,6-trihydroxyhexane
bis(2-ethylhexyl)adipate	1-methyl-2-pyrrolidinone
1,2-propane diol	ethylene glycol
2-ethyl-2(hydroxymethyl)-1,3-propane diol	2-ethoxyethyl acetate

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Table 2.
MTBE Extractable Compounds

2,2,4,4-Tetramethyl-1,3-pentanone
2-Ethoxyethyl acetate
2-Ethoxy ethanol
2,6-Dimethyl-4-heptanol
2-Ethyl-1-hexanol
Dimethyl malonate
Hexanoic acid
2,2,4-Trimethyl-1,3-pentane diol
2-Ethyl hexanoic acid
Heptanoic acid
Octanoic acid
bis(2-Ethylhexyl)adipate

Table 3.
Aqueous Extract (after MTBE extraction) Compounds

1,2-propane diol
ethylene glycol
1-methyl-2-pyrrolidinone
1,3-butane diol
2,2-dimethyl-1,3-propane diol
dipropylene glycol
1,4-butane diol
diethylene glycol
1,6-hexane diol
e-caprolactam
triethylene glycol
cis,trans-1,4-cyclohexane dimethanol
2-ethyl-2(hydroxymethyl)-1,3-propane diol
1,2,6-trihydroxyhexane

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Table 4.
Gas Chromatograph Conditions
for MTBE Extract Analysis

Gas Chromatograph	Hewlett-Packard Model 5880 with Model 3396 Integrator
Detector	Flame Ionization Detector (FID)
Column	DB-FFAP (polyethylene glycol - acid modified) fused silica capillary, 30 m x 0.53 i.d., 1.0 um film thickness
Injection Volume	3 uL (splitless)
Injection Port Liner	Uniliner (Restek)
Carrier Gas	Helium
Linear Velocity	18 cm/sec (head pressure 4 psi)
Initial Temperature	60°C
Initial Time	1 minute
Oven Temperature Rate	4°C/minute
Final Temperature	230°C
Final Temperature Hold	15 minutes
Detector Temperature	250°C
Detector Makeup Gas	Nitrogen
Makeup Gas Flow Rate	30 ml/minute
Detector Attenuation	Attn 2 ↑ 0

Table 5.
Gas Chromatograph Conditions
for Aqueous (after MTBE Extraction) Analysis

Gas Chromatograph	Hewlett-Packard Model 5880 with Model 3396 Integrator
Detector	Flame Ionization Detector (FID)
Column	DB-WAX (polyethylene glycol) fused silica capillary, 30 m x 0.53 i.d., 1.0 um film thickness
Injection Volume	3 uL (splitless)
Injection Port Liner	Uniliner (Restek)
Carrier Gas	Helium
Linear Velocity	18 cm/sec (head pressure 4 psi)
Initial Temperature	60°C
Initial Time	1 minute
Oven Temperature Rate	4°C/minute
Final Temperature	220°C
Final Temperature Hold	15 minutes
Detector Temperature	250°C
Detector Makeup Gas	Nitrogen
Makeup Gas Flow Rate	30 ml/minute
Detector Attenuation	Attn 2 ↑ 0

Table 6.
Selected Ketone, Glycol and Diol
MTBE Extractable Compounds
Validation Results

Parameter	Spike	Percent Recovery									Mean	%
		0.1 ppm			1 ppm			10 ppm				
	Run	1	2	3	1	2	3	1	2	3	% Rec	RSD
2,2,4,4-tetramethyl-3-pentanone		36	42	35	63	53	59	74	81	81	58	29
2-ethoxethyl acetate		0	0	0	6	6	6	6	7	6	4	NC
2-ethoxy ethanol		65	81	88	74	75	74	71	76	75	76	8.0
2,6-dimethyl-4-heptanol		79	98	97	94	100	97	110	114	110	100	10
2-ethyl-1-hexanol		94	114	122	103	110	108	116	121	119	112	8.5
dimethyl malonate		56	69	76	63	63	62	61	66	65	65	8.1
hexanoic acid		76	54	59	82	97	97	100	120	106	100	21
2,2,4-trimethyl-1,3-pentane diol		58	0	0	70	62	51	23	24	21	34	NC
2-ethyl hexanoic acid		100	94	107	89	107	105	107	124	112	107	9.6
heptanoic acid		97	85	97	88	102	101	104	123	109	105	11
octanoic acid		113	104	123	88	107	105	107	130	113	109	11
bis-(2-ethylhexyl) adipate		109	115	115	123	137	135	128	139	136	126	11

NC is defined as not calculated.

Table 7.
Selected Ketone, Glycol and Diol
Aqueous Concentrate Compounds
Validation Results

Parameter	Spike	Percent Recovery									Mean	%
		0.1 ppm			1 ppm			10 ppm				
	Run	1	2	3	1	2	3	1	2	3	% Rec	RSD
1,2-propane diol		86	83	96	110	105	115	99	98	100	99	9.6
ethylene glygol		80	87	79	110	100	112	96	95	97	95	12
1-methyl-2-pyrrolidinone		105	98	109	99	103	107	96	94	97	101	5.0
1,3-butane diol		97	91	101	112	112	116	99	97	101	103	7.8
2,2-dimethyl-1,3-propane diol		96	101	102	93	92	97	87	85	90	94	5.8
dipropylene glycol		87	95	97	116	113	117	115	103	103	105	11
1,4-butane diol		91	99	97	120	114	119	111	107	111	108	9.5
diethylene glycol		82	88	84	112	106	115	104	102	104	100	12
1,6-hexane diol		87	93	93	109	104	110	101	97	102	100	7.2
e-caprolactam		102	113	107	107	101	107	103	101	103	105	3.7
triethylene glycol		94	98	88	111	103	113	109	104	109	103	8.0
cis/trans-1,4-cyclohexane dimethanol		91	94	96	83	80	83	82	79	81	85	7.1
cis/trans-1,4-cyclohexane dimethanol		102	105	104	87	84	87	84	82	85	91	10
2-ethyl-2(hydroxymethyl)-1,3-propane diol		0	0	0	121	117	129	115	115	120	120	4.9
1,2,6-trihydroxy hexane		0	0	0	91	89	101	102	104	108	100	7.4

NC is defined as not calculated.

Appendix 3

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Analytical Results of
Selected Ketone, Glycol and Diol Compounds
in Groundwater Samples
from the Hooker/Ruco Superfund Site
Hicksville, New York
May 4, 1992

SUMMARY

Groundwater samples from six monitoring wells at Hooker/Ruco Superfund Site at Hicksville, New York were analyzed for selected ketone, glycol, and diol compounds. These compounds were water soluble compounds utilized as raw materials at this site (see Table 1).

The groundwater samples from monitoring wells P1, F1, J1, 10593, K1, and L1 were collected on 11/06 and 11/07/91. The samples were extracted on 11/07 and 11/08/91 with methyl tert-butyl ether (MTBE) and both the MTBE extract and the remaining aqueous sample were concentrated and analyzed for the selected compounds using flame ionization gas chromatography.

The major component determined in the groundwater samples was 2,2-dimethyl-1,3-propane diol in concentrations ranging from 0.10 to 220 mg/L. Other compounds determined were 2,6-dimethyl-4-heptanol, 2,2,4-trimethyl-1,3-pentane diol, hexanoic acid, 2-ethyl-hexanoic acid, octanoic acid, diethylene glycol, ethylene glycol, dipropylene glycol and triethylene glycol. Selected compounds not detected in groundwater samples were: 2,2,4,4-tetramethyl-3-pentanone; 2-ethyl-1-hexanol; dimethyl malonate; hexanoic acid; heptanoic acid; octanoic acid; bis(2-ethylhexyl) adipate; 1,2-propane diol; 1-methyl-2-pyrrolidinone; 1,3-butane diol; 1,4-butane dio; 1,6-hexane diol; e-caprolactam; cis/trans-1,4-cyclohexane dimethanol; 2-ethyl-2(hydroxymethyl-1,3-propane diol and 1,2,6-trihydroxy hexane.

INTRODUCTION

Groundwater samples were collected from six wells identified as P1, F1, J1, 10593, K1, and L1 from the Hooker/Ruco Superfund Site in Hicksville, New York. The groundwater samples were collected in duplicate into 1 Liter amber glass bottles with teflon lined caps.

EXPERIMENTAL

The groundwater samples were analyzed for selected organic compounds using the methodology described in ***Analytical Method for the Analysis of Selected Ketone, Glycol and Diol Compounds in Ground Water, April 27, 1992.***

The samples were also analyzed for Total Organic Carbon (TOC) using SW-846, 3rd Edition Method 9060 on 11/11/91.

RESULTS AND DISCUSSION

The results of the analyses of the groundwater samples for selected ketone, glycol and diol compounds which were extracted into MTBE are contained in Tables 2. Samples P1, F1, J1, 10593, and L1 contained concentrations of some of the selected ketone, glycol and diol compounds greater than 0.1 mg/L. Sample F1 contained the highest total concentration of selected semi-volatile compounds. The MTBE extract from sample L1 did not contain any of the selected semi-volatile organic compounds (<0.10 mg/L). Residual amounts of 2,2-dimethyl-1,3-propane diol were present (below the quantification limit) in the MTBE extracts, due to the high concentrations present in the aqueous concentrates.

The results from the analysis of aqueous concentrates of groundwater samples which had been extracted with MTBE are contained in Table 3. The aqueous concentrates from ground water samples collected from all six well locations had measurable concentrations of the selected ketone, glycol and diol compounds. The major constituent determined was 2,2-dimethyl-1,3-propane diol.

A summary of the compounds analyzed in the groundwater samples collected from six wells along with the respective TOC content for each sample are contained in Table 4. The concentrations of compounds determined were converted to their carbon equivalent to enable comparison to reported TOC concentrations. The percentage of carbon accounted for by the selected ketone, glycol and diol compounds ranged from 10 to 86%

QA/QC

The performance of the analytical method was determined by spiking both blank MilliQ water and groundwater samples with known concentrations of the selected ketone, glycol and diol compounds. Percent recovery of selected ketone, glycol and diol compounds extracted into MTBE are contained in Table 5. The recoveries of selected compounds fortified at 0.1, 1.0 and 10 ppm in MilliQ water and groundwater were comparable and ranged from 44 to 90%.

Percent recovery of ketone, glycol and diol compounds determined in the aqueous concentrate are contained in Table 6. MilliQ water and groundwater samples which were fortified at 0.1, 1.0, 4.0 and 10 ppm had average recoveries ranging from 53 to 113%. The recovery of the selected compounds from MilliQ water and groundwater samples were comparable, except for 2-ethyl-2(hydroxymethyl)-1,3-propane diol and 1,2,6-trihydroxy hexane for which lower recoveries were obtained from the groundwater samples.

The performance of the TOC analyses was determined by spiking both a blank deionized water blank and a groundwater sample. The percent recovery of the DI blank spike and the sample spike were: 110% and 98% respectively. A reference standard was analyzed containing 400 mg/L carbon. The percent recovery of the reference standard was 106%. The results are shown in Table 7.

Table 1.
Selected Ketone, Glycol, and Diol Compounds

2,2,4,4-tetramethyl-1,3-pentanone	1,3-butane diol
2-ethoxy ethanol	2,2-dimethyl-1,3-propane diol
2,6-dimethyl-4-heptanol	dipropylene glycol
2-ethyl-1-hexanol	1,4-butane diol
dimethyl malonate	diethylene glycol
hexanoic acid	1,6-hexane diol
2,2,4-trimethyl-1,3-pentane diol	e-caprolactam
2-ethyl hexanoic acid	triethylene glycol
heptanoic acid	cis,trans-1,4-cyclohexane dimethanol
octanoic acid	1,2,6-trihydroxyhexane
bis(2-ethylhexyl)adipate	1-methyl-2-pyrrolidinone
1,2-propane diol	ethylene glycol
2-ethyl-2(hydroxymethyl)-1,3-propane diol	2-ethoxyethyl acetate

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Table 2.
MTBE Extract
Analytical Results
mg/L

Parameter	Date	P1	F1	J1	10593	K1	L1
		11/06/91	11/06/91	11/06/91	11/07/91	11/07/91	11/07/91
2,2,4,4-tetramethyl-3-pentanone		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
2,6-dimethyl-4-heptanol		ND 0.1	1.3	0.1	ND 0.1	ND 0.1	ND 0.1
2-ethyl-1-hexanol		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
dimethyl malonate		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
hexanoic acid		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
2,2,4-trimethyl-1,3-pentane diol		0.2	1.1	0.3	ND 0.1	ND 0.1	ND 0.1
2-ethyl-hexanoic acid		0.4	4.0	0.1	ND 0.1	ND 0.1	ND 0.1
heptanoic acid		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
octanoic acid		ND 0.1	ND 0.1	ND 0.1	ND 0.1	0.1	ND 0.1
bis(2-ethylhexyl) adipate		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
2,2-dimethyl-1,3-propane diol		0.5	31	1.5	0.3	0.1	ND 0.1

ND x is defined as not detected at or above x.

Table 3.
Aqueous Concentrate
Analytical Results
mg\L

Parameter		P1	F1	J1	10593	K1	L1
	Date	11/06/91	11/06/91	11/06/91	11/07/91	11/07/91	11/07/91
1,2-propane diol		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
ethylene glygol		ND 0.1	ND 0.1	0.1	0.1	0.1	ND 0.1
1-methyl-2-pyrrolidinone		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
1,3-butane diol		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
2,2-dimethyl-1,3-propane diol		5.3	190	4.9	4.3	2.1	0.1
dipropylene glycol		ND 0.1	ND 0.1	ND 0.1	0.1	ND 0.1	ND 0.1
1,4-butane diol		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
diethylene glycol		ND 0.1	ND 0.1	ND 0.1	0.3	0.2	0.1
1,6-hexane diol		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
e-caprolactam		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
triethylene glycol		ND 0.1	ND 0.1	ND 0.1	0.2	0.1	0.1
cis/trans-1,4-cyclohexane dimethanol		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
cis/trans-1,4-cyclohexane dimethanol		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
2-ethyl-2(hydroxymethyl)-1,3-propane diol		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1
1,2,6-trihydroxy hexane		ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1	ND 0.1

ND x is defined as not detected at or above x.

Table 4.
Total Organic Carbon Results

Parameter	P1	P1	F1	F1	J1	J1	10593	10593	K1	K1	L1	L1
	ppm	ppmC	ppm	ppmC	ppm	ppmC	ppm	ppmC	ppm	ppmC	ppm	ppmC
2,2-dimethyl-1,3-propane diol	5.8	3.4	220	130	6.4	3.7	4.6	2.7	2.2	1.3	0.1	0.06
2,6-dimethyl-4-heptanol			1.3	0.98	0.1	0.08						
2,2,4-trimethyl-1,3-pentane diol	0.2	0.13	1.1	0.73	0.32	0.21						
2-ethyl hexanoic acid	0.42	0.28	4	2.6	0.11	0.07						
octanoic acid			0.1	0.07								
hexanoic acid			0.1	0.06								
ethylene glycol							0.1	0.04	0.1	0.04		
diethylene glycol							0.33	0.15	0.2	0.09	0.13	0.06
triethylene glycol							0.2	0.1	0.1	0.05	10.12	0.06
dipropylene glycol							0.1	0.05				
Total	6.4	3.8	220	130	6.9	4.1	5.3	3	2.6	1.4	0.48	2.4
TOC (ppm)		13		190		26		6.2		4		10
Percent as Total (ppmC)/TOC (ppm)		28		68		26		86		36		

ppm is defined as parts per million
ppmC is defined as ppm of carbon (mole ratio)

Table 5.
MTBE Extract
Spike Recovery Data
Percent Recovery (%)

Parameter	Blank Spike 0.1 ppm	Blank Spike 10 ppm	K1 Spike 1 ppm	K1 Spike 1 ppm	Blank Spike 0.1 ppm	Blank Spike 1 ppm
2,2,4,4-tetramethyl-3-pentanone	42	76	49	46	22	66
2,6-dimethyl-4-heptanol	73	92	75	73	60	79
2-ethyl-1-hexanol	82	92	79	77	69	81
dimethyl malonate	48	52	41	40	39	46
hexanoic acid	107	91	81	82	100	91
2,2,4-trimethyl-1,3-pentane diol	59	87	57	61	56	72
2-ethyl-hexanoic acid	60	91	82	86	57	76
heptanoic acid	66	88	77	81	60	74
octanoic acid	69	88	81	92	63	75
bis(2-ethylhexyl) adipate	89	100	85	96	88	92

Table 6.
Aqueous Concentrate
Spike Recovery Data
Percent Recovery (%)

Parameter	Blank Spike 0.1 ppm	Blank Spike 10 ppm	K1 Spike 1 ppm	K1 Spike 1 ppm	Blank Spike 1 ppm	K1 Spike 4 ppm	K1 Spike 1 ppm	10593 Spike 1 ppm
1,2-propane diol	89	101	80	82	110	116	133	117
ethylene glygol	97	104	67	71	123	139	151	152
1-methyl-2-pyrrolidinone	84	96	80	81	91	94	96	80
1,3-butane diol	83	95	74	81	100	104	110	94
2,2-dimethyl-1,3-propane diol	93	106	81	89	90	105	138	117
dipropylene glycol	86	102	73	78	142	124	116	112
1,4-butane diol	88	95	103	113	101	106	100	96
diethylene glycol	95	90	98	97	74	82	93	119
1,6-hexane diol	80	92	72	70	90	99	85	80
e-caprolactam	82	94	66	62	95	96	77	78
triethylene glycol	106	91	36	35	106	152	88	80
cis/trans-1,4-cyclohexane dimethanol	70	76	55	55	107	105	88	99
cis/trans-1,4-cyclohexane dimethanol	76	79	54	54	110	109	72	67
2-ethyl-2(hydroxymethyl)-1,3-propane diol	0	50	16	15	92	84	57	57
1,2,6-trihydroxy hexane	0	47	20	19	121	114	26	30

Table 7.
Total Organic Carbon
Spike Recovery Data

Identification	Amount Added mg/L	Amount Found mg/L	Spike Recovery %
Blank Spike	1000	1200	120%
Sample F1 Spike	1	1.1	110%
Reference Standard	400	420	106%